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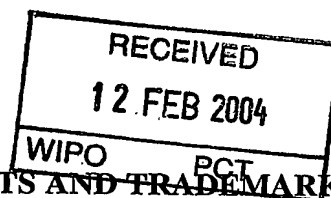
**February 09, 2004**

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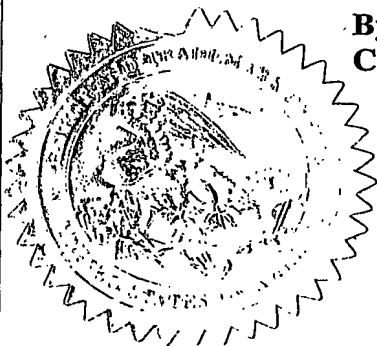
**APPLICATION NUMBER: 60/428,958**

**FILING DATE: November 25, 2002**

**RELATED PCT APPLICATION NUMBER: PCT/US03/37894**



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60420950 112306

**PROVISIONAL APPLICATION FOR PATENT COVER SHEET**

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c)

JC997 U.S. PTO



11/25/02

Docket No. 02400-0180P (4338-265005)

**INVENTOR(s)**

LAST NAME	FIRST NAME	M.I.	RESIDENCE (city & either state or foreign country)
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10872 U.S. P. 60/428958

**TITLE OF THE INVENTION (280 characters max)****SOLID FILTRATION MEDIA WITH ENHANCED PORE STRUCTURE****CORRESPONDENCE ADDRESS**

23370

PATENT TRADEMARK OFFICE

CUSTOMER NUMBER: 23370

APPLY BAR CODE  
LABEL HERE →**ENCLOSED APPLICATION PARTS (check all that apply)**☒ Specification Number of Pages 28☐ Drawing(s) Number of Sheets☐ Other (specify)☒ Provisional Application Filing Fee**METHOD OF PAYMENT**

- ☒ A check is enclosed to cover the Provisional Application filing fee.
- ☒ Applicant claims Small Entity status.
- ☒ The Commissioner is hereby authorized to charge any additional filing fee and credit any refund to Deposit Account No. 11-0855.

FILING FEE: \$ 80

The invention was not made by an agency of the U.S. Government nor under a contract with an agency of the U.S. Government.

Respectfully submitted,

SIGNATURE:

Date: November 25, 2002

TYPED OR PRINTED NAME: David E. Wigley, Ph.D.

Reg. No. 52,362

☐ Additional inventors are being named on separately numbered sheets attached hereto.**"Express Mail" Mailing Label Number EL910742452US**

**FEE TRANSMITTAL**

Attorney Docket No. 02400-0180P (45038-265005)

This sheet accompanies a patent application transmittal for the following application:

Inventor(s): **WILLIAM G. ENGLAND**Filing Date: **November 25, 2002**Title: **SOLID FILTRATION MEDIA WITH ENHANCED PORE STRUCTURE**

The filing fee is calculated as shown below:

**1. FILING FEE:**

FOR:	SMALL ENTITY		LARGE ENTITY	
	FEE	FEE PAID	FEE	FEE PAID
<input type="checkbox"/> UTILITY FILING FEE	\$370		\$740	
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<input checked="" type="checkbox"/> PROVISIONAL FILING FEE	\$80	\$80	\$160	
SUBTOTAL (1)		\$80		\$

**2. CLAIMS:**

FOR:	SMALL ENTITY			LARGE ENTITY	
	NO. FILED	NO. EXTRA	RATE	FEE	RATE
TOTAL CLAIMS	- 20 =		x 9 =		x 18 =
INDEP. CLAIMS	- 3 =		x 42 =		x 84 =
<input type="checkbox"/> MULTIPLE DEPENDENT CLAIM PRESENTED			+140 =		+280 =
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**3. ADDITIONAL FEES:**

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<input type="checkbox"/> LATE FILING, FEE OR OATH	\$65		\$130	
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Reg. No. 52,362Date: 25 November 2002

## SOLID FILTRATION MEDIA WITH ENHANCED PORE STRUCTURE

5

### TECHNICAL FIELD

The present invention relates generally to compositions and methods for the removal of compounds having toxic or corrosive properties or disagreeable odors, especially sulfur containing compounds, from gaseous streams. The invention more particularly relates to methods for controlling and improving the pore structure of a solid filtration medium used for the removal of undesirable compounds from gaseous streams.

### 15 BACKGROUND OF THE INVENTION

Undesirable airborne compounds, including sulfur compounds, ammonia, formaldehyde, urea, carbon monoxide, oxides of nitrogen, mercaptans, amines, and ethylene, occur in a number of environments, where most are primarily responsible for the presence of disagreeable odors, or irritating or toxic gases. Such environments include petroleum treatment and storage areas, sewage treatment facilities, hospitals, morgues, anatomy laboratories, animal rooms, and pulp and paper production sites, among others. These undesirable compounds may be bacterial breakdown products of higher organic compounds, or simply byproducts of industrial processes.

Hydrogen sulfide ("H<sub>2</sub>S"), a colorless, toxic gas with a characteristic odor of rotten eggs, is produced in coal pits, gas wells, sulfur springs, and from decaying organic matter containing sulfur. Controlling emissions of this gas, particularly from municipal sewage treatment plants, has long been considered desirable. More recently, protecting electronic apparatus from the corrosive fumes of these compounds has become increasingly important. Further, H<sub>2</sub>S is flammable.

Ammonia (" $\text{NH}_3$ "), also a colorless gas, possesses a distinctive, pungent odor and is a corrosive, alkaline gas. The gas is produced in animal rooms and nurseries and its control also has long been considered desirable.

5 Chlorine (" $\text{Cl}_2$ ") is a greenish-yellow gas with a suffocating odor. The compound is used for bleaching fabrics, purifying water, treating iron, and other uses. Control of this powerful irritant is most desirable for the well-being of those who work with it or are otherwise exposed to it. At lower levels, in combination with moisture, chlorine has a corrosive effect on electronic circuitry, stainless steel and the like.

10 Formaldehyde (" $\text{OCH}_2$ ") is a colorless gas with a pungent, suffocating odor. It is present in morgues and anatomy laboratories, and because it is intensely irritating to mucous membranes, its control is desirable.

Urea (" $\text{OC}(\text{NH}_2)_2$ ") is present in toilet exhaust and is used extensively in the paper industry to soften cellulose. Its odor makes control of this compound desirable.

15 Carbon monoxide (" $\text{CO}$ "), an odorless, colorless, toxic gas, is present in compressed breathing air. Oxygenation requirements for certain atmospheres, including those inhabited by humans, mandate its control.

Oxides of nitrogen, including nitrogen dioxide (" $\text{NO}_2$ "), nitric oxide (" $\text{NO}$ "), and nitrous oxide (" $\text{N}_2\text{O}$ "), are compounds with differing characteristics and levels of danger to humans, with nitrous oxide being the least irritating oxide. Nitrogen dioxide, however, is a deadly poison. Control of pollution resulting from any of these oxides is desirable or necessary, depending on the oxide.

20 Mercaptans and amines, including methyl mercaptan (" $\text{CH}_3\text{SH}$ "), butyl mercaptan (" $\text{C}_4\text{H}_9\text{SH}$ ") and methyl amine (" $\text{CH}_3\text{NH}_2$ "), are undesirable gases present in sewerage odor. The control of these gases is desired for odor control.

25 Ethylene (" $\text{C}_2\text{H}_4$ ") is a colorless, flammable gas. It is a simple asphyxiant that accelerates the maturation or decomposition of fruits, vegetables, and flowers. Control of this compound prolongs the marketable life of such items.

30 Attempts have been made to provide solid filtration media for removing the undesirable compounds listed above from fluid streams. Desired features of such media are a high total capacity for the removal of the targeted compound, a high

efficiency in removing the compound from an air stream contacting the media, and a high ignition temperature (non-flammability). High capacity and high efficiency are, in turn, directly affected by the porosity and pore structure of the solid filtration media, while the capacity, efficiency and ignition temperature are all affected by the specific composition of the media.

A solid filtration media for the removal of undesirable compounds from gas streams is described in U.S. Patent No. 4,235,750, which discloses an apparatus and method for adsorbing ethylene and other gaseous contaminants. The apparatus is a three-part container in which permanganate impregnated alumina is present in one compartment, activated carbon in the second compartment, and a mixture of molecular sieves and activated silica gel in the third compartment. The '750 patent limits the concentration of the aqueous potassium permanganate to one pound of permanganate dissolved in one gallon of water. This patent teaches that, if more is dissolved, the pores of the alumina become clogged when impregnated with the permanganate, thereby reducing its oxidizing capacity. Therefore optimal concentration of potassium permanganate in the dried, finished product of the '750 patent is about 4 to 5%, preferably 4.5% of potassium permanganate by weight of the finished product. This filtration media would not be useful in small filter beds because large quantities of the impregnated alumina are required to compensate for its limited capacity.

Another example of a solid oxidizing system in pellet form consisting of activated alumina impregnated with potassium permanganate is described in U.S. Patent No. 3,049,399. The pellets disclosed in the '399 patent provide air purification and odor control by both adsorbing and absorbing odors, and then destroying the collected odors using the controlled oxidizing action of the potassium permanganate. As described in the '750 patent above, the alumina of the '399 patent is impregnated with a 5% aqueous solution of the permanganate and subsequently dried. Therefore, the pellets of the '399 patent also exhibit a limited capacity for removing undesired contaminants from gas streams.

Yet another example of a solid filtration media for removing undesirable compounds from a gas stream is disclosed in U.S. Patent No. 3,226,332. The '332

patent discloses a method of producing granular activated alumina uniformly impregnated with a solid oxidizing agent, preferably potassium permanganate, for use in treating fluid streams. The impregnate solution is sprayed onto a dry combination being tumbled in a mixer thereby forming pellets which are later dried to remove a  
5 substantial portion of the remaining water. The preferred range of potassium permanganate taught in the '332 patent is from about 2% to about 4%. Accordingly, these pellets would be expected to have a limited capacity for the removal of undesirable compounds from fluid streams, and would demonstrate the same shortcomings as the potassium impregnated alumina of the '750 patent discussed  
10 above.

Although there are a variety of permanganate-impregnated substrates known for removing undesirable contaminants from fluid streams, as demonstrated above, these known impregnated substrates all demonstrate a limited capacity for the removal of undesirable compounds from gas streams. These limitations arise to a  
15 large extent from the insufficient porosity of the solid filtration media that results in these media not meeting the needs of various industries.

Therefore, what is needed is a high efficiency, high capacity, low flammability permanganate impregnated substrate for the removal of undesirable compounds from gas streams. Such an impregnated substrate needs to be long-lasting, requiring fewer  
20 replacements and thereby minimizing replacement and maintenance costs. Also needed is a high capacity impregnated substrate that may be used in small filter beds, and therefore may allow the treatment of fluid streams where there are significant space limitations.

## 25 SUMMARY OF THE INVENTION

A solid filtration media and a method of preparing the solid filtration media, method of controlling and enhancing the pore structure of a solid filtration medium, and method of treating a fluid stream with the solid Filtration media are provided. The solid filtration media of this invention is useful for removing or reducing  
30 undesirable contaminants from a gaseous fluid stream.

The solid filtration media is a porous impregnated substrate having high levels of impregnate. The filtration media preferably contains permanganate, water, a porous substrate such as activated alumina, and a gas-evolving or gas-producing material such as sodium bicarbonate. In contrast to the presently available filtration media, the high capacity media described herein contains levels of permanganate greater than 5% while exhibiting a pore structure that is controlled and enhanced by the amount of the gas-evolving material contained in the media. The pore structure is improved or enhanced by providing an increase in either the pore size or the number of viable pores in the filtration media. As a result of having permanganate levels greater than 5% and the enhanced porosity, the media provides an increased efficiency for removing compounds such as hydrogen sulfide from gaseous streams. For example, the media described herein has a hydrogen sulfide capacity of greater than approximately 15%, whereas currently available media are known to have a maximum hydrogen sulfide capacity less than 10%.

In accordance with the method of producing the filtration media described herein, a porous substrate is impregnated with an aqueous permanganate solution in the presence of a gas-evolving or gas-producing material. The concentration of gas-evolving material in the media mixture directly affects the resulting porosity of the solid filtration media, and in turn, its efficiency and capacity. The capacity of the filtration media described herein is equivalent to that provided when using activated carbon adsorbents. However, the filtration media of the present invention is less expensive and considerably less flammable than activated carbon adsorbents. Due to its high capacity for adsorbing and treating undesirable compounds, the media is long-lasting, thereby minimizing maintenance and replacement costs. The high capacity filtration media is useful in small filter beds, and therefore may allow the treatment of fluid streams where significant space limitations exist.

Generally described, the filtration media provided herein contains at least approximately 5% permanganate salt, at least about 5% water, at least about 5% of a gas-evolving material, and a porous substrate, wherein these percentages are by weight of the composition. Preferably, the permanganate salt is potassium permanganate. The gas-evolving material is usually selected from a carbonate



compound, a bicarbonate compound, or a combination thereof, that function by producing a gas (in this case,  $\text{CO}_2$ ) upon heating. The porous substrate is selected from activated alumina, silica gel, a zeolite, adsorbent clay, kaolin, activated bauxite, or combinations thereof, the preferred porous substrates being alumina or an alumina-zeolite mix.

Preferably, the improved solid filtration media of the present invention comprises between approximately 5 and 12% permanganate salt, between approximately 5 and 30% gas-evolving material, between approximately 5 and 35% water, and a porous substrate, wherein the above percentages are by weight of the composition. As the ambient humidity varies, the concentration of water in the composition varies as well, with 30-35% by weight of the composition constituting water under high humidity conditions.

The present invention also provides a method of preparing an improved filtration media composition. This method comprises the steps of mixing water, a permanganate salt, a gas-evolving material, and a substrate, and then forming the mixture into at least one cohesive porous unit. The unit is then cured at a temperature of from about  $100^\circ\text{F}$  to about  $200^\circ\text{F}$ , until the concentration of water is at least about 5% by weight of the composition but most preferably between about 10 to about 25% by weight, the concentration of the permanganate salt is at least about 5% by weight of the composition but most preferably between about 7 and about 12% by weight, and the concentration of the gas-evolving material is at least about 5% by weight of the composition but most preferably between about 15 and about 20% by weight. This process is usually accomplished by forming a solid mixture described above, spinning the solid mixture on a pelletizing disk at an angle (typically  $45^\circ$ ) while spraying water onto the solid mixture, which forms the mixture into at least one cohesive porous unit. The cohesive units are then cured at temperature of from about  $100^\circ\text{F}$  to about  $200^\circ\text{F}$ , until the desired composition with respect to water and permanganate composition are attained.

Also provided herein is a method of controlling the pore structure of a solid filtration media. In this regard, the curing step is also crucial in controlling and enhancing the porosity of the composition, because the gas-evolving material is

selected so as to release a gaseous compound during this step, by any mechanism. The evolution of gas is responsible for producing voids and spaces in the solid filtration media during curing, thereby enhancing pore structure. Moreover, the extent of pore structure may be dictated by, among other factors, the relative amount or weight percent of gas-evolving material present in the solid media mixture. By  
 5 controlling porosity or pore structure, it is meant principally increasing the size of existing pores, although increasing the number of viable pore, for example by modifying pore size distribution, is also included in this definition. All of these factors affect the amount of  $\text{KMnO}_4$  that can be incorporated in the media and  
 10 therefore the capacity to chemisorb undesirable compounds. For example, increasing the amount of gas-evolving material present in the solid media mixture increases the size of and number of pores in the resulting solid filtration media.

In one embodiment, the methods of the present invention encompass forming an aqueous solution containing the permanganate salt and gas-evolving material and  
 15 then mixing this aqueous solution with the porous substrate. In an alternative aspect of the invention, the methods entail forming a dry mixture comprising the permanganate salt, a gas-evolving material, and the substrate, and then adding water to the dry mixture. In yet another aspect of the invention, the methods of this invention involve forming a dry mixture comprising the permanganate salt, the  
 20 substrate, and optionally the gas-evolving material, forming an aqueous solution comprising the permanganate salt and optionally the gas-evolving material, and then mixing the aqueous solution with the dry mixture. Thus, the gas-evolving material such as sodium bicarbonate may be added either to the dry mixture, to the water, or to both in the method of preparing the filtration media.

25 Preferably, in the method of the present invention, the unit formed is cured until the concentration of water is from about 5 to about 35% by weight of the composition, most preferably from about 10 to about 25% by weight. Preferably, the concentration of the permanganate salt is from about 5 to about 12% by weight of the composition, and the concentration of sodium bicarbonate is between about 5 to 30%  
 30 by weight. Most preferably, the concentration of the permanganate salt is from about

7 to about 12% by weight of the composition, and the concentration of sodium bicarbonate is between about 15 to 20% by weight.

Yet another aspect of the solid filtration media is a method of treating a contaminated fluid stream with the improved solid filtration composition of the present invention. This method entails contacting the contaminated fluid stream with the improved solid filtration composition that has been produced using the porosity control method outlined above.

The improved filtration media embodying the present invention, the pore structure control method of its preparation, and the method of its use provide the desired improved efficiency and capacity in removing contaminants from gas streams.

Accordingly, it is an object of the solid filtration media to provide an improved solid filtration media having a high efficiency and high total capacity for removing undesirable compounds from an air stream.

It is another object to provide a method of controlling the pore structure of a solid filtration media and thereby control and enhance the efficiency and capacity of the media for removing unwanted compounds from an air stream.

It is also an object of the solid filtration media to provide an improved solid filtration media that is relatively inexpensive to manufacture and use.

It is yet another object to provide a method of making an improved solid filtration media having a high efficiency and a high total capacity for the removal of an undesirable compound.

It is a further object of the solid filtration media to provide a method of treating a contaminated air or gas stream with an improved solid filtration media.

These and other objects, features and advantages of the present invention will become apparent after a review of the following detailed description of the disclosed embodiments and the appended claims.

#### DETAILED DESCRIPTION OF THE INVENTION

The present invention relates to an improved solid filtration media and method of preparing the same, method of controlling and enhancing the pore structure of a solid filtration media, and method of treating a fluid stream with the solid filtration

media. The filtration media can be used to remove or reduce undesirable contaminants from a gaseous fluid stream. The improved solid filtration media contains permanganate, water, a gas-evolving material such as sodium bicarbonate, and a porous substrate such as activated alumina. In some embodiments, at least one  
5 zeolite is optionally included in the solid filtration media. The media contains significantly higher levels of permanganate and water than previously thought possible, in part because of the method provided herein for controlling and enhancing the pore structure of the media. An improved efficiency of removal of compounds is therefore achieved by this media. For example, a preferred embodiment of this  
10 improved media has a hydrogen sulfide capacity of greater than approximately 15%, whereas the media produced by the current art has a maximum hydrogen sulfide capacity of approximately 9-10%.

Generally described, the present invention provides an improved filtration media comprising a substrate impregnated with high levels of both permanganate and  
15 water, and uses variations in the weight percent of a gas-evolving material, typically sodium bicarbonate, to adjust the pore structure of the media to the desired level. The filtration media comprises at least approximately 5% permanganate salt, at least about 5% water, and at least about 5% gas-evolving material, by weight of the composition, after curing. The permanganate salt may be selected from, but not limited to,  
20 potassium permanganate ( $\text{KMnO}_4$ ), sodium permanganate, magnesium permanganate, calcium permanganate, barium permanganate, lithium permanganate, or combinations thereof. Preferably, the permanganate salt is potassium permanganate (Aldrich Chemical Company, Milwaukee, Wisconsin). The concentration of the potassium permanganate in the solid filtration media is preferably between approximately 5 and  
25 12%, and most preferably between 7 and 12%, after curing.

The gas-evolving material of the filtration media described herein is a material that produces or releases a gaseous substance upon heating, for example during the curing step of forming the filtration media. The bubbles formed in this heating process are instrumental in enhancing and controlling the pore structure of the  
30 filtration media. The gas-evolving material is usually selected from a carbonate compound, a bicarbonate compound, or a combination thereof, that function by

producing carbon dioxide gas upon heating. A preferred gas-evolving material is sodium bicarbonate, because of its smooth release of carbon dioxide, and its relatively low cost. However, other bicarbonates and carbonates can be used in this invention, the selection of which is understood by one of ordinary skill in the art. The number and size of the pores produced from heating the gas-evolving material is related to the concentration of the gas-evolving material in the solid filtration media, the temperature of curing, and the time of curing. Thus, increasing the concentration of sodium bicarbonate in the composition increases the pore size and number, and helps reduce and prevent clogging of the pore structure, and enhance the retention of water and sustain the concentration of the permanganate in the filtration media.

Examples 1 and 2 below provide specific details of how to prepare a solid filtration media composition of this invention, and Example 3 illustrates several compositions of permanganate-impregnated substrates that are prepared using the methods described in Examples 1 and 2. Example 4 provides an alternative method of preparing a potassium permanganate and sodium bicarbonate impregnated alumina composition that complements the methods of Examples 1 and 2. These examples also serve illustrate how to improve the pore structure of a sample of potassium permanganate and sodium bicarbonate impregnated alumina pellets by controlling the sodium bicarbonate concentration. The capacity of solid filtration media of the present invention to chemisorb undesirable air-borne contaminants increases as the percentage of gas-evolving material, in this case sodium bicarbonate, in the solid filtration increases. During curing, the increased evolution of gas from the higher concentration of gas-evolving materials such as sodium bicarbonate is presumed to be responsible for producing larger voids and spaces in the solid filtration media, thereby enhancing pore structure. Therefore, the extent of pore structure is affected by, among other factors, the relative amount or weight percent of gas-evolving material present in the solid media mixture. For example comparing Sample 3A and Sample 3D from Example 3, Sample 3A with its higher concentration of sodium bicarbonate provides a solid filtration media with increased porosity and pore structure. By controlling or increasing porosity or pore structure, it is meant principally the

increasing the size of existing pores, although increasing the number of viable pore, for example by modifying pore size distribution, is also included in this description.

The selection of the porous substrate for use in the solid filtration media also affects the properties, including the pore size distribution, in the final filtration media.

5 The porous substrate may be selected from the group consisting of, but not limited to, activated alumina ( $\text{Al}_2\text{O}_3$ ) (UOP Chemical, Baton Rouge, Louisiana), silica gels (J. M. Huber, Chemical Division, Havre De Grace, Maryland), zeolites (Steel Head Specialty Minerals, Spokane, Washington), kaolin (Englehard Corp., Edison, New Jersey), adsorbent clays (Englehard Corp., Edison, New Jersey), and activated  
10 bauxite. One preferred porous substrate is alumina. Preferably, the concentration of substrate in the filtration media is between 40 and 80%, and most preferably between, 40 and 60%.

Another preferred porous substrate is a combination of alumina and a zeolite, in which up to about 50% by weight of the porous substrate combination is a zeolite.  
15 It is believed that zeolites further control the moisture content of the filtration media by attracting and holding water, which functions to keep more of the impregnate in solution. This effect, in turn, is believed to provide the filtration media with its higher capacity and improved efficiency. As used herein, the term zeolite includes natural silicate zeolites, synthetic materials and phosphate minerals that have a zeolite-like  
20 structure. Examples of zeolites that can be used in this media include, but are not limited to, amicitite (hydrated potassium sodium aluminum silicate), analcime (hydrated sodium aluminum silicate), pollucite (hydrated cesium sodium aluminum silicate), boggsite (hydrated calcium sodium aluminum silicate), chabazite (hydrated calcium aluminum silicate), edingtonite (hydrated barium calcium aluminum silicate),  
25 faujasite (hydrated sodium calcium magnesium aluminum silicate), ferrierite (hydrated sodium potassium magnesium calcium aluminum silicate), gobbinsite (hydrated sodium potassium calcium aluminum silicate), harmotome (hydrated barium potassium aluminum silicate), phillipsite (hydrated potassium sodium calcium aluminum silicate), clinoptilolite (hydrated sodium potassium calcium aluminum  
30 silicate), mordenite (hydrated sodium potassium calcium aluminum silicate), mesolite (hydrated sodium calcium aluminum silicate), natrolite (hydrated sodium aluminum

silicate), amicitite (hydrated potassium sodium aluminum silicate), garronite (hydrated calcium aluminum silicate), perialite (hydrated potassium sodium calcium strontium aluminum silicate), barrerite (hydrated sodium potassium calcium aluminum silicate), stilbite (hydrated sodium calcium aluminum silicate), thomsonite (hydrated sodium calcium aluminum silicate), and the like. Zeolites have many related phosphate and silicate minerals with cage-like framework structures or with similar properties as zeolites, which may also be used in place of, or along with, zeolites. These zeolite-like minerals include minerals such as kehoeite, pahasapaite, tiptopite, hsianghualite, lovdarite, viseite, partheite, prehnite, roggianite, apophyllite, gyrolite, maricopaite, okenite, tacharanite, tobermorite, and the like.

The concentration of water in the filtration media is preferably between approximately 5 and 35%, most preferably between 10 and 25%. One of ordinary skill in the art will understand that the concentration of free water in the filtration media may be altered by the conditions present, such as the humidity and the temperature, during its storage and use.

Preferably, the improved solid filtration media of the present invention contains between approximately 5 and 12% potassium permanganate, between approximately 5 and 30% sodium bicarbonate, between approximately 5 and 35% water, and between approximately 40 and 80% alumina, by weight of the final composition. Most preferably, the improved solid filtration media comprises between approximately 7 and 12% potassium permanganate, between approximately 15 and 20% sodium bicarbonate, between approximately 10 and 25% water, and between approximately 40 and 60% alumina, by weight.

It is to be understood that when referring to the relative weight of components, the water referred to in the present specification, Examples, and Tables is defined as the free water, and does not include the bound water in the substrate. Free water is driven off by an oven at approximately 200°F, but if left in the substrate it is available for the oxidation reaction. In contrast, bound water is not driven out or evaporated except by a kiln at 1800 to 2000°F, and the bound water functions by holding the substrate together. Bound water is not available for reaction with the undesirable contaminants.

It is also to be understood that the term permanganate in the present specification, examples, and tables represents the permanganate salt, not the permanganate ion  $\text{MnO}_4^-$ . Therefore, the percent ranges of permanganates in compositions in the present specification denote the percent of the permanganate salt in the composition, not the percent of the permanganate ion in the composition.

Terms such as "filtration media", "adsorbent composition," "chemisorbent composition," and "impregnated substrate" are all interchangeable, and denote a substance that is capable of reducing or eliminating the presence of unwanted contaminants in fluid streams by the contact of such a substance with the fluid stream. It is to be understood that the term "fluid" is defined as a liquid or gas capable of flowing, and includes gaseous, aqueous, organic containing, and inorganic containing fluids.

The present invention also provides a method of preparing an improved solid filtration composition. This method includes mixing water, a permanganate salt, a gas-evolving material, and a porous substrate, and then forming the mixture into at least one cohesive porous unit. The unit is then typically cured at a temperature of from about 100°F to about 200°F, until the concentration of water is at least about 5% by weight of the composition, and the concentration of the permanganate salt is at least about 7% by weight of the composition.

The size and shape of the cohesive porous unit is not critical to the present invention. Any size and shape of a porous unit known in the art to reduce or eliminate undesirable contaminants from fluid streams when in contact with the unit may be used in the present invention. Preferably, the porous unit is a nominal 1/8" diameter round pellet.

The method of the present invention preferably includes forming an aqueous solution containing the permanganate salt and gas-evolving material and then mixing the aqueous solution with the porous substrate. To dissolve and maintain the permanganate salt in solution, the aqueous solution should be heated to approximately 160° to 200°F, and preferably to between approximately 180° to 190°F.

In another embodiment of the invention, the method includes forming a dry mixture containing the permanganate salt, the gas-evolving material, and the porous



substrate, and then adding water to the dry mixture. In yet another embodiment of the invention, the method includes forming a dry mixture containing the permanganate salt, optionally the gas-evolving material, and the substrate, forming an aqueous solution containing the permanganate salt, optionally the gas-evolving material, and then mixing the aqueous solution with the dry mixture. Thus, the gas-evolving material such as sodium bicarbonate may be added either to the dry mixture, to the water, or to both in the above methods of preparing the filtration media.

Typically, the solid filtration media is produced by the following process. First, a solid mixture containing potassium permanganate, a gas-evolving material, and a porous substrate in the desired proportions is prepared and mixed. This solid mixture is added to a spinning pelletizing disk while spraying water onto the solid mixture to form it into at least one cohesive porous unit. The pelletizing disk is typically spinning at an angle of about  $45^\circ$  relative to the ground, and this angle can be adjusted to dictate the size of the individual cohesive units that are formed in the process. Finally, curing the units at a temperature of from about  $100^\circ\text{F}$  to about  $200^\circ\text{F}$  is effected, until sufficient water is evaporated to achieve the desired concentration of components.

Preferably, in the method of the present invention, the unit formed is cured until the concentration of water is from about 5 to about 35%, most preferably between about 10 and 25% by weight of the composition; the concentration of the permanganate salt is from about 5 to about 12% by weight of the composition, most preferably between about 7 and 12%; and the concentration of the gas-evolving material is from about 5 to about 30% by weight of the composition, most preferably between about 15 and 20% by weight of the composition, after curing. The presence of sodium bicarbonate allows for a lower curing temperature, such as  $130^\circ$  to  $140^\circ\text{F}$ , in contrast to the conventional curing temperature of about  $200^\circ\text{F}$ .

The impregnation treatment of the activated starting material in accordance with the present invention has not been found to be critical with respect to the particular sequence in which the dry mix is impregnated with moisture and impregnates. The above combinations may be mixed in any manner which

effectively produces the desired filtration media. Impregnation may be carried out simply by immersing and soaking the solid combination in a volume of impregnate solution. Also, the impregnate solution may be passed through the combination rather than being used as a static immersion treatment. However, it has been found that a preferred method of impregnation is spray addition in which an impregnate solution is sprayed onto a dry combination being tumbled in a mixer. This method of impregnation has been described in U.S. Pat. No. 3,226,332, which is herein incorporated by reference. Other methods of impregnating the combinations would suggest themselves as equally appropriate, and these are included within the scope of the present invention.

In one embodiment utilizing the above spray addition method, the aqueous impregnate solution of potassium permanganate is sprayed onto a dry combination of sodium bicarbonate and a porous substrate, such as activated alumina. For example, the dry combination would contain between approximately 80 to 85% activated alumina, and between approximately 15 to 20% of sodium bicarbonate.

The concentration of the potassium permanganate may vary in the solution to be sprayed onto the dry combination. For example, to produce a solid filtration medium containing approximately 7% potassium permanganate, an aqueous solution containing approximately 20% of potassium permanganate, at between approximately 160°F to 200°F, and preferably at about 180°F to 190°F should be sprayed on the dry combination of gas-evolving material and porous substrate being tumbled in a mixer. Also, to produce a solid filtration medium containing approximately 10% potassium permanganate, a solution of approximately 30% potassium permanganate at between approximately 160°F to 200°F, and preferably at about 180°F to 190°F should be sprayed on the dry combination of gas-evolving material and porous substrate being tumbled in a mixer. Any concentration of permanganate in the aqueous solution which is effective to yield the composition of the present invention may be used. Further, where the permanganate salt is either in the dry feed mixture or in both the aqueous solution and the dry feed mixture, any concentration of permanganate in the dry mixture and/or the aqueous solution which is effective to produce the composition

of the present invention may be used. For example, the media may be used to fill perforated modules to be inserted into air ducts in a manner known in the art.

Yet another aspect of the present invention is a method of treating a contaminated fluid stream using the improved solid filtration composition of the present invention, produced by the process described above. This method involves contacting the contaminated fluid stream with the solid filtration composition described above. Typically, the undesired contaminants will be removed from air, especially from air admixed with effluent gas streams resulting from municipal waste treatment facilities, paper mills, petrochemical refining plants, morgues, hospitals, anatomy laboratories, and hotel facilities, and so forth. Methods of treating gaseous or other fluid streams are well known in the art.

The composition of the present invention is useful for removing undesired compounds from gaseous streams. The concentrations of these undesirable compounds in the gaseous streams is not considered critical to the process of the present invention, nor is the physical and chemical makeup of the gas stream from which it is desired to remove undesirable compositions considered critical. Even concentrations of these undesirable compounds in gas streams resulting in levels lower than one ppb of the compounds passing through a solid filtration media bed per minute can be removed.

However, it has been found that flow rates of the gas stream being contacted with the bed of filtration media affect the breakthrough capacities of the media. The preferred flow rate is between 10 and 750 ft./min., and most preferably is between 60 and 100 ft./min., flowing perpendicularly to the face of the bed.

While not intending to be bound by the following statement, it is believed that it may be necessary that certain oxidizing conditions prevail while using the solid filtration media. The extent of oxidation may affect the degree of purification achieved. Preferably, oxygen is present in the gas stream being treated, at least in small amounts. This oxygen content is readily found in the gas stream, if air constitutes a sufficient portion of the gas stream being treated. If oxygen is totally absent or present in insufficient amounts, oxygen may be independently introduced into the gas stream being treated. A number of factors affect the amount of oxygen,

which may be required for maximum removal of the contaminants in a gas stream in accordance with the present invention, including the concentration and absolute amount of compounds being removed from the gas stream being treated.

5       With respect to the amount of compound removed, it is believed that the following factors affect the process: the basic degree of attraction of the activated substrate for the compound; the pore structure and size of the substrate; the specific surface area of the substrate as affected by the number and size of pores; the surface characteristics of the substrate; the amount of permanganate present; the amount of gas-evolving material present in the composition, which affects the number, size, and  
10       perhaps structure of pores; and the amount of water present.

      The filtration media of the present invention is appropriately used alone in filter beds for the removal of undesirable compounds. It is also appropriate, however, to use the composition of the present invention in conjunction with filter beds containing other filtration media, and also in conjunction with mechanical or  
15       electrostatic filters. Any such additional filters may be placed either upstream (before the media of the present invention with respect to the effluent gas being treated) or downstream.

      The above methods, including the method for controlling pore structure, significantly increase the efficiency and capacity of impregnated porous substrates  
20       (filtration media) to remove certain undesired compounds from gaseous streams over the capacity of impregnated substrates currently available. Therefore, the lifetime of a specific quantity of the improved filtration media will be much longer than the same quantity of the currently available filtration media. The extension of the lifetime of the filtration products will significantly reduce the purchasing, servicing, and  
25       installation costs of consumers and businesses. Also, the enhanced efficiency of the improved media allows for a new line of products which are compact versions of currently available units, but have the same performance as the larger, currently available units. The capability of creating significantly smaller filtration units is useful for providing efficacious air filtration in space-limited quarters which  
30       previously could not utilize the larger, currently available units.

Also, the improved permanganate media is less expensive than other filtration media having a roughly equivalent capacity. For example, the media of the present invention has a capacity equivalent to the contaminant capacity of activated carbon adsorbents. However, the media of the present invention is considerably less  
5 expensive than activated carbon adsorbents.

Further, the filtration media of the present invention is safe as it is not flammable, in contrast to carbon-containing filtration products. This aspect of the present invention is significant to industries that manufacture or process flammable, fume producing materials, such as the petroleum industry for example.

10 In media of the present invention, the increase in the concentration of the potassium permanganate and water in the solid filtration media, along with the ability to control and increase the pore structure through sodium bicarbonate addition, have greatly increased the media's capacity for removing contaminants from air streams. As described earlier, the currently available potassium permanganate impregnated  
15 alumina media has a capacity for the removal of hydrogen sulfide of approximately 9-10%. In contrast, the composition described herein containing approximately 53% activated alumina, 23% water, 7% potassium permanganate, and 17% sodium bicarbonate, has a hydrogen sulfide capacity of approximately 15 to 17%.

The results of accelerated efficacy tests comparing the capacity of solid  
20 filtration media of the prior art and the solid filtration media described in this invention demonstrate the improved efficacy of the latter. For example, a commercially available media, which contained approximately 3.5-4.0% potassium permanganate, and 10-15% water, had a hydrogen sulfide capacity of approximately 7.5%. In contrast, the solid filtration media of the present invention, which contained  
25 approximately 7% potassium permanganate, 15-20% sodium bicarbonate, and 15-20% water, had a hydrogen sulfide capacity of 15.9%. The efficacy tests were performed by challenging a known quantity of the selected solid filtration media with 1.0% hydrogen sulfide gas at a constant flow rate and monitoring the concentration of hydrogen sulfide in the gas stream exiting the solid filtration media. The accelerated  
30 efficacy test is fully described in U.S. Patent No. 6,004,522, which is herein incorporated by reference.

The results of a non-accelerated test for the determination of performance characteristics such as removal efficiency and capacity for removal of various gas-phase air filtration media also demonstrate the improved performance for the filtration media of this invention. The efficiency tests were performed on a solid filtration media of the prior art, and on a solid filtration media of the present invention. The media of the prior art contain 4.4% potassium permanganate, 19.9% water, 17% sodium bicarbonate, and 58.7% alumina. The media of the present invention contain 8.4% potassium permanganate, 24.4% water, 17% sodium bicarbonate, and 50.2% alumina. The media of the present invention has a percent efficiency of hydrogen sulfide removal of 96% at 260 hours, 78% at 456 hours, and 54% at 600 hours. In contrast, the media of the prior art has a hydrogen sulfide removal efficiency of approximately 92% at 260 hours, 42% at 456 hours, and 15% at 600 hours. The non-accelerated efficacy test is also fully described in U.S. Patent No. 6,004,522, which is incorporated by reference.

Although the applicant does not fully appreciate the precise mechanisms by which the improved media operates, and is not bound by the following theory, it is believed that the oxidation reactions between the permanganate and the undesirable contaminants occur primarily near the surface of the filtration media, rather than deep within its pores. Therefore it is probably critical that the surface's oxidative capabilities be continually regenerated. It is believed that the oxidative capability of the surface of the media is regenerated by the flow or migration of permanganate from the center of the media to the surface of the media while the products of the oxidation reactions flow or migrate from the surface of the media to the center of the media. It is also believed that the higher the concentration of permanganate at the surface of the media, the higher the capacity and efficiency of the media.

Further, the fluidity of the permanganate solution directly affects the flow and thus the quantity of the permanganate reaching the surface of the media. Therefore, it is critical to maintain an elevated concentration of free water in the media so that the permanganate solution maintains a high level of fluidity and readily flows to the surface of the media thereby maximizing the efficiency and capacity of the media. A liquid path thus should be established between the interior of the pores and the surface

of the media. In this regard, the improved pore structure provided by the addition of a gas-evolving material to the filtration media is believed to enhance the ready flow of permanganate solution. This is contrary to conventional theories, which teach a need for penetration of the gaseous contaminants into the pores of the substrate.

5       The new theory, presented above, explains why the capacity and efficiency of the currently available filtration media could not surpass the capacity and efficiency obtained at the potassium permanganate concentrations of 4-5% with the filtration media described herein. As stated above, previously, various attempts were made to impregnate the media with higher quantities of potassium permanganate, however, the  
10 majority of the free water has always been removed from these media. As stated above, the efficiency and capacity of the highly impregnated media currently available remains constant or decreases relative to the capacity achieved by media impregnated with 4-5% permanganate and with enhanced pore structure due to sodium bicarbonate use.

15       The following examples will serve better to illustrate the composition, the treatment methods of the present invention, and the capacity for the removal of contaminants in gas streams produced thereby. It should be noted that the continuous flow systems described in several of the following examples all were operated at a relative humidity of 40-50%.

20

#### EXAMPLE 1

##### *Preparation of a Solid Filtration Media Composition*

A 7% potassium permanganate and sodium bicarbonate impregnated alumina composition was prepared as follows.

25       A dried feed mix was prepared by combining, by weight, 80 to 85% alumina, and 15 to 20% sodium bicarbonate. The dry feed mixture was sprayed with a heated aqueous potassium permanganate solution at 180 to 190°F while the dried feed mix was tumbled in a tumble mill. The resulting pellets were then dried at 130 to 140°F until the pellets contained about 20 to 25% free water.

30       To prepare solid filtration media containing approximately 7% potassium permanganate by dry weight, the aqueous potassium permanganate solution

preferably contained approximately 20% potassium permanganate by weight. It is to be understood that the aqueous potassium permanganate solution was sprayed onto the dry feed mix while the dry mix was rolled in the pelletizing disk as described in U.S. Patent No. 3,226,332, incorporated herein by reference.

5

## EXAMPLE 2

### *Preparation of a Solid Filtration Media Composition*

A 10% potassium permanganate and sodium bicarbonate impregnated alumina composition was prepared as follows.

10 A dried feed mix was prepared by combining, by weight, 80 to 85% alumina, and 15 to 20% sodium bicarbonate. The dry feed mixture was sprayed with a heated aqueous potassium permanganate solution at 180 to 190°F while the dried feed mix was being tumbled in a tumble mill. The resulting pellets were then dried at 130 to 140°F in air until the pellets contained about 20 to 25% free water.

15 To prepare a solid filtration media containing approximately 10% potassium permanganate, by dry weight, the aqueous solution preferably contained approximately 25% potassium permanganate, by weight. It is to be understood that the aqueous potassium permanganate solution was sprayed on to the dry feed mix while the dry mix was rolled in the pelletizing disk as described in U.S. Patent No.  
20 3,226,332, incorporated herein by reference.

## EXAMPLE 3

### *Compositions of Permanganate Impregnated Substrates*

Using the method described in Example 1 or Example 2, the following  
25 compositions, by dry weight, were also prepared.



TABLE I. Composition of Solid Filtration Media

Sample Number	Substrate	% Substrate	% NaHCO <sub>3</sub>	% H <sub>2</sub> O	% KMnO <sub>4</sub>
3A	Alumina	50	18	20	12
3B	Alumina	50	20	20	10
3C	Alumina	50	20	22	8
3D	Alumina	65	8	15	12
3E	Alumina	75	0	15	10
3F	Silica gel	50	18	22	10
3G	Zeolite	55	10	23	12
3H	Adsorbent Clay	55	13	20	12

The dry feed mix was prepared by mixing appropriate amounts of the substrate and the sodium bicarbonate together. The dry feed mix was mixed in a tumbling mill with an appropriate amount of an aqueous potassium permanganate solution sprayed onto the dry feed mix while tumbling, in the manner described in U.S. Patent No. 3,226,332. Curing was carried out as in Examples 1 or 2 to provide the cured pellets as a strong, non-dusting filter media suitable for placement in filter beds, which provided an efficient media for the reduction or elimination of many undesirable compounds.

#### EXAMPLE 4

##### *Alternative method of preparing potassium permanganate and sodium bicarbonate impregnated alumina composition*

A dry feed mix is prepared by combining, by weight, 75 to 85% alumina, 7 to 12% finely divided potassium permanganate, and 10 to 20% sodium bicarbonate. The dry feed mixture is sprayed with water while the dried feed mix is being tumbled in a tumble mill. The resulting pellets are then dried at 130° to 140°F until the pellets contain about 20 to 25% free water. It is to be understood that the dry feed mix is rolled in the pelletizing disc as described in U.S. Patent No. 3,226,332, while it is being sprayed with water. It is also to be understood that the dry feed mix may contain less potassium permanganate, and the water which is sprayed on the dry feed

mix may contain the corresponding amount of potassium permanganate to yield the same final concentration of potassium permanganate as when all of the potassium permanganate is in the dry feed mix. If such a method is utilized, the aqueous potassium permanganate solution preferably is to be heated to approximately 180° to 190°F.

It should be understood, of course, that the foregoing relates only to certain embodiments of the present invention and that numerous modifications or alterations may be made therein without departing from the spirit and the scope of the invention. All of the publications or patents mentioned herein are hereby incorporated by reference in their entireties.

## CLAIMS

What is claimed is:

- 5           1.     A method of controlling the pore structure of a solid filtration composition, comprising adjusting the weight percent of a gas-evolving material in the composition,
- wherein the solid filtration composition consists essentially of at least approximately 5% potassium permanganate by weight of the composition; at least  
10     approximately 5% water by weight of the composition; at least approximately 5% of a gas-evolving material by weight, and a porous substrate.
2.     The method of Claim 1, wherein the gas-evolving material is selected from a carbonate compound, a bicarbonate compound, or a combination thereof.
- 15           3.     The method of Claim 1, wherein the porous substrate is selected activated alumina, silica gels, zeolites, zeolite-like minerals, kaolin, adsorbent clays, activated bauxite, or a combination thereof.
- 20           4.     The method of Claim 1, wherein the gas-evolving material is sodium bicarbonate and the porous substrate is activated alumina or a combination of activated alumina and at least one zeolite or zeolite-like mineral.
- 25           5.     The method of Claim 4, wherein the zeolite or zeolite-like mineral is selected from amicite, analcime, pollucite, boggsite, chabazite, edingtonite, faujasite, ferrierite, gobbinsite, harmotome, phillipsite, clinoptilolite, mordenite, mesolite, natrolite, garronite, perlialite, barrerite, stilbite, thomsonite, kehoeite, pahasapaite, tiptopite, hsianghualite, lovdarite, viseite, partheite, prehnite, roggianite, apophyllite, gyrolite, maricopaite, okenite, tacharanite, tobermorite, or a combination thereof.
- 30

6. The method of Claim 4, wherein the concentration of sodium bicarbonate is between approximately 5 and 30% by weight of the composition.

7. The method of Claim 4, wherein the concentration of sodium bicarbonate is between approximately 15 and 20% by weight of the composition.

8. The method of Claim 1, wherein the concentration of potassium permanganate is between approximately 5 and 12% by weight of the composition.

9. The method of Claim 1, wherein the concentration of potassium permanganate is between approximately 7 and 12% by weight of the composition.

10. The method of Claim 1, wherein the water concentration is between about 5% and 35%.

11. The method of Claim 1, wherein the water concentration is between about 10% and 25%.

12. A method of treating a contaminated fluid stream, comprising contacting the contaminated fluid stream with a solid filtration composition such that the contaminants are removed from the contaminated fluid stream,

wherein the solid filtration composition is produced by the process of: forming a solid mixture consisting essentially of potassium permanganate, a gas-evolving material, and a porous substrate; spinning the solid mixture on a pelletizing disk at an angle while spraying water onto the solid mixture, thereby forming the mixture into at least one cohesive porous unit; and curing the unit at a temperature of from about 100°F to about 200°F, until the concentration of water is at least approximately 5% by weight of the composition, the concentration of gas-evolving material is at least approximately 5% of a by weight, and the concentration of potassium permanganate is at least about 5% by weight of the composition.

13. The method of Claim 12, wherein the gas-evolving material is selected from a carbonate compound, a bicarbonate compound, or a combination thereof.

14. The method of Claim 12, wherein the porous substrate is selected  
5 activated alumina, silica gels, zeolites, zeolite-like minerals, kaolin, adsorbent clays, activated bauxite, or a combination thereof.

15. The method of Claim 12, wherein the gas-evolving material is sodium  
bicarbonate and the porous substrate is activated alumina or a combination of  
10 activated alumina and at least one zeolite or zeolite-like mineral.

16. The method of Claim 15, wherein the zeolite or zeolite-like mineral is  
selected from amicitite, analcime, pollucite, boggsite, chabazite, edingtonite, faujasite,  
ferrierite, gobbinsite, harmotome, phillipsite, clinoptilolite, mordenite, mesolite,  
15 natrolite, garronite, perialite, barrerite, stilbite, thomsonite, kehoeite, pahasapaite,  
tiptopite, hsianghualite, lovdarite, viseite, partheite, prehnite, roggianite, apophyllite,  
gyrolite, maricopaite, okenite, tacharanite, tobermorite, or a combination thereof.

17. The method of Claim 15, wherein the unit is cured until the  
20 concentration of sodium bicarbonate is between approximately 5 and 30% by weight  
of the composition.

18. The method of Claim 15, wherein the unit is cured until the  
concentration of sodium bicarbonate is between approximately 15 and 20% by weight  
25 of the composition.

19. The method of Claim 12, wherein the unit is cured until the  
concentration of potassium permanganate is between approximately 5 and 12% by  
weight of the composition.

30

20. The method of Claim 12, wherein the unit is cured until the concentration of potassium permanganate is between approximately 7 and 12% by weight of the composition.

5 21. The method of Claim 12, wherein the unit is cured until the water concentration is between about 5% and 35%.

22. The method of Claim 12, wherein the unit is cured until the water concentration is between about 10% and 25%.

10

## ABSTRACT

An improved filtration media and method of preparing the media, method of controlling the pore structure of solid filtration media, and method of treating a fluid stream with the media are provided. In one embodiment, the improved media comprises a substrate having elevated levels of permanganate and water, and engineered by adjusting the amount of a gas-evolving component such as sodium bicarbonate, so as to provide enhanced control over the porosity of the filtration media during its manufacture. More specifically, the improved media comprises a porous substrate, impregnated with at least approximately 5% permanganate, and at least approximately 5% water, at least approximately 5% gas-evolving material, all by weight.

Docket No.: 02400-0180P (45038-265005)

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